

MECHANICAL AND THERMAL PROPERTIES OF MONTMORILLONITE
FILLED DATE PALM LEAF FIBER REINFORCED RECYCLED
POLYETHYLENE TEREPHTHALATE NANOCOMPOSITES

ALIREZA DEGHANI

UNIVERSITI TEKNOLOGI MALAYSIA

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You will forgive me, my dear father, if I set your name, so dear to me, at the head of an insignificant brochure. I am too impatient to await another opportunity of giving you a small proof of my love.

I want to dedicate this thesis to my precious wife, Sara. Without her help and encouragement it simply never would have been.

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ABSTRACT

Date palm leaf fiber (DPLF) reinforced recycled poly(ethylene terephthalate) (PETr) nanocomposites containing montmorillonite (MMT) were prepared by melt extrusion using a counter rotating twin-screw extruder followed by injection molding. The influence of the DPLF and MMT along with 10 phr of SEBS-g-MA as compatibilizer on the mechanical and thermal properties of the PETr matrix was evaluated separately, through their individual contributions. The effect of various DPLF additions at from 5 to 15 wt% and the incorporation of 1, 3 and 5 phr of MMT were investigated. Scanning electron microscopy (SEM) was used to investigate the phase morphology and study the adhesion between the matrix and DPLF fibers while the dynamic mechanical properties were studied via dynamic mechanical analysis (DMA). The thermal properties were determined using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The results showed that blending SEBS-g-MA with PETr matrix significantly increased the toughness at the expense of stiffness of the blend. The incorporation of DPLF resulted in enhancements in tensile and flexural strength of the composites. However, a decrease in the Young's and flexural moduli was recorded. Fiber additions also improved the impact strength of the composites and an increase in the area under the stress-strain curve was observed. SEM revealed a strong interfacial bonding between the matrix and fibers, and a homogenous one phase system, indicating strong interactions between the PETr matrix and SEBS-g-MA. The DSC results showed that the crystallization process was enhanced through the incorporation of DPLF fibers, and a higher degree of crystallinity was observed as compared to PETr. However, TGA showed that, fiber addition lowered the thermal stability of the composites. The incorporation of MMT resulted in a significant increase in the stiffness of the nanocomposites and 58% increment was observed in the flexural modulus was recorded. The addition of nanoparticles also increased the degree of crystallinity for 1 and 3 phr of nanoclay addition. Thermal stability enhancements were observed for all nanocomposite formulations. The results pointed to a successful development of a hybrid nanocomposite from recycled PET and natural fiber with the potential of various outdoor applications.

ABSTRAK

Gentian daun pokok kurma (GDPK) diperkukuh dengan nanokomposit poli(etilena terephthalate) (PETr) yang dikitar semula terisi montmorillonit (MMT) telah disediakan melalui penyemperitan leburan menggunakan penyemperitan skru berkembar berlawanan arah dan diikuti dengan pengacuan suntikan. Sifat-sifat mekanikal dan terma terhadap GDPK, MMT bersama 10 phr stirena-etilina-butadiena-stirena tercangkuk malik anhidrida (SEBS-g-MA) yang digunakan sebagai penyerasi ke atas PETr matrik telah di kaji secara berasingan melalui ciri-ciri bagi setiap bahan. Kesan variasi penambahan GDPK dari 5 sehingga 15% berat dan penambahan MMT sebanyak 1, 3 dan 5 phr terhadap campuran telah dikaji. Mikroskop imbasan electron (SEM) telah digunakan untuk mengkaji fasa morfologi permukaan dan interaksi di antara matrik dan gentian GDPK manakala sifat dinamik mekanikal telah dikaji melalui analisis dinamik mekanikal (DMA). Sifat terma pula telah ditentukan menggunakan analisa thermogravimetri (TGA) dan kalorimeter pembezaan imbasan (DSC). Keputusan yang diperolehi menunjukkan bahawa pengadunan SEBS-g-MA bersama PETr matrik telah meningkatkan ketahanan tetapi menyebabkan penurunan kekakuan campuran tersebut. Campuran GDPK telah meningkatkan kekuatan regangan dan lenturan komposit manakala modulus Young dan modulus lenturan menunjukkan penurunan. Penambahan gentian telah meningkatkan kekuatan hentaman komposit dan penambahan lengkung tegasan-terikan telah diperhatikan. SEM telah menunjukkan bahawa terdapat ikatan antara permukaan yang kuat di antara matrik dan gentian, dan satu fasa sistem yg sekata yang membuktikan bahawa interaksi yang kuat diantara PETr matrik dan SEBS-g-MA. Keputusan DSC menunjukkan bahawa proses penghabluran telah meningkat melalui penambahan gentian GDPK dan penambahan darjah penghabluran telah diperhatikan berbanding PETr. Bagaimanapun, keputusan TGA menunjukkan dengan penambahan gentian di dalam komposit telah menurunkan kestabilan terma komposit. Penambahan MMT ke dalam komposit telah meningkatkan kekakuan dengan ketara dan peningkatan sebanyak 58% bagi modulus lenturan telah diperhatikan. Penambahan nanopartikel telah meningkatkan darjah penghabluran bagi 1 dan 3 phr dengan penambahan nanotananah liat. Peningkatan kestabilan terma telah diperhatikan bagi kesemua formulasi nanokomposit.

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LIST OF ABBREVIATIONS

ASTM	–	American Society for Testing and Materials
BHET	–	Bis (hydroxyethyl) terephthalate
DMA	–	Dynamic Mechanical Analysis
DMT	–	Dimethyl Terephthalate
DPLF	–	Date Palm Leaf Fiber
DSC	–	Differential Scanning Calorimeter
EPA	–	Environmental Protection Agency
E'	–	Storage Modulus
FTIR	–	Fourier transform infrared spectroscopy
HIPP	–	High Impact Polypropylene
Hz	–	Hertz
ISS	–	Interfacial shear strength
LDPE	–	Low Density Polyethylene
LLDPE	–	Linear Low Density Polyethylene
MAH	–	Maleic Anhydride
MFI	–	Melt flow index
MMT	–	Montmorillonite
NF	–	Natural Fiber
NFRC	–	Natural Fiber Reinforced Composites
OMLS	–	Organically modified layered silicates
PA6	–	Polyamide 6
PC	–	Polycarbonate
PE	–	Polyethylene
PET	–	Poly(ethylene terephthalate)
PETr	–	Recycled Poly(ethylene terephthalate)
PLA	–	Poly(lactic acid)
PS	–	Polystyrene

PTT	–	Poly(trimethylene terephthalate)
SEBS-g-MA	–	Anhydride-grafted poly(styrene-ethylene/butyl-diene-styrene)
SEM	–	Scanning electron microscopy
TEM	–	Transmission electron microscopy
TGA	–	Thermogravimetric analysis
T_c	–	Crystallization temperature
T_d	–	Decomposition temperature
T_g	–	Glass transition temperature
T_m	–	Melting temperature
X_c	–	Degree of crystallinity

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CHAPTER 1

INTRODUCTION

1.1 Background

Poly (ethylene terephthalate) (PET) has been used extensively due to its interesting physical and mechanical properties. It is a light weight, transparent thermoplastic with good barrier properties, making it suitable for packaging applications. Its widespread use in the packaging and beverage industry, results in a huge number of post consumed PET bottles and makes it a very good target for polymer recycling [1].

The amount of plastics end up in city landfills is increasing yearly and they usually end up in incinerators and an effective recycling program is necessary to address this issue. A study by Environmental Protection Agency (EPA) showed that almost one fifth of the total waste stream are plastic wastes [2]. The annual world PET production is around 60 million tons, which were manufactured using extrusion, injection molding and blow molding techniques [3].

Studies showed that during the reprocessing of PET, the melt flow index increased from 23 to 80 g/10 min for virgin PET and for the material after five processing cycles [4]. An increase in the concentration of the carboxyl groups due to chemical and mechanical degradations was also reported [4]. After five times of injection molding, the chain packing was facilitated by the presence of smaller chains that tended to fit among the larger ones. Hence, the degree of crystallization increased from 23% to 37%. A ductile to brittle transition was observed during the reprocessing of PET. Virgin resin showed a ductile behavior and had more than 40% of elongation at break where recycled PET only shows 5% due to changes in degree of crystallinity.

Similar brittleness problems have been solved before by incorporating a tough material into a brittle matrix. Huang [5] examined the rubber toughening of an amorphous polyamide using maleic anhydride-grafted poly(styrene-ethylene/butyl-diene-styrene) (SEBS-g-MA) and SEBS in terms of morphology, Izod impact behavior and ductile to brittle behavior transition temperature changes. A super tough nylon 66 was successfully produced by blending it with SEBS-g-MA [6]. Several studies have also been conducted recently in the development of toughened nanocomposites [7–9]. Tjong [8] had successfully developed and studied a toughened polypropylene/montmorillonite (MMT) nanocomposite by the incorporation of SEBS-g-MA through dynamic mechanical analysis (DMA). More recently a toughened polycarbonate (PC) nanocomposite was developed with enhanced ductility and impact strength [9]. Despite all these efforts, very few studies focused on the toughening effect of SEBS-g-MA addition on the PETr matrix. Zhang [10] had examined the effect of SEBS-g-MA addition as compatibilizer into the PETr/LLDPE blend and reported enhancements in tensile and impact strengths as well as elongation at break. The SEBS-g-MA addition improved the Izod impact strength and elongation at break of PETr while lowering the stiffness of the blend [11]. Interestingly, the incorporation of SEBS-g-MA into the PETr matrix increased the crystallization rate and overall degree of crystallinity and resulted in the formation of smaller crystal entities [12].

Natural fibers are promising alternatives for traditional reinforcing components in polymer composites due to their low cost, abundance availability, biodegradability, high specific strength and low density [13–15]. Natural fiber reinforced polymer composites may offer a new class of materials, which can provide environmental protection as well as significant advantages (in terms of reduced composite weight and biodegradability) over mineral reinforcements such as glass fiber, mica and talc. In addition, there are several important features of natural fibers such as: nonabrasive nature, low energy consumption, biodegradability, high specific properties, availability of a wide variety of fibers throughout the world, and generation of agriculture based economy [14]. All these benefits imply a significant property potential for commodity synthetic polymers. Therefore, development of natural fiber reinforced recycled thermoplastic composites offers materials with balanced properties while equally reducing the volume of plastic waste.

Natural fiber reinforced composites (NFRC) have been studied extensively due to the environmentally aware consumers, increased environmental pollution and global warming [14, 16–20]. Natural fibers (NF) as reinforcing elements in the composites are sourced from renewable resources, which will reduce the cost and

the dependence on petroleum. Today, natural fiber reinforced composites are used in a variety of applications where low to medium strength and low cost are needed. These applications may vary from door and window frames and furnitures to car door panels. Several parts in commercial vehicles, particularly those produced in Brazil, use natural fibers such as coconut coir, sisal and flax and epoxy resin in order to achieve higher performance due to their superior impact resistance in addition to noise reduction [15, 21].

The date palm tree naturally grows in the middle east, north Africa and countries like India, Iraq, Iran, Pakistan and also in the United States. There are greater than one hundred types of date palm trees in the world and each tree can grow for more than one hundred years [19]. The large number of date palm tree throughout the world produces a huge amount of waste, which was considered unusable before [24]. The fibers produced from the date palm tree annually, creates a very consistent source of natural fiber, which can be used in the composite industry.

One main task was to modify the natural fibers in such a way that these fibers be more compatible with the hydrophobic polymer matrices. In the previous study by Valadez [18], 5% NaOH solution was used to modify the henequen fiber surface and much stronger interactions between the fiber and the HDPE matrix was observed when the fiber's surface topography was altered. In a more recent study, flax fibers were surface treated to increase the effectiveness of the compatibilizer and also to make the fibers more compatible with the PP matrix and reported lower water absorptions when alkali surface treatment was used [22]. Fiber surface modification parameters should be optimized in order to achieve the desired properties and prevent fiber damage. A complete discussion on the effects of surface treatment methods on mechanical and thermal properties of date palm fiber by Al-Khanbashi [23] revealed that using too abrasive chemicals and long exposure times could damage the fiber's surface and reduce its tensile strength. Between commercial detergent, 5% NaOH solution and dioxin solution, surface treatment with low concentration sodium hydroxide solution showed enhancements in thermal resistance and Young's modulus as well as tensile strength of the fibers [24].

1.2 Problem statement

It is known that the incorporation of natural fibers into synthetic polymers helps to improve the mechanical properties. However, these fibers possess hydrophilic character and blending them with polar hydrophobic polymers raises an issue of incompatibility. Previous study by Corradini [1] showed that the addition of sugar bagasse fiber into recycled PET (PETr) did not yield satisfying mechanical properties. In order to overcome this problem, the simultaneous use of surface modifications and compatibilizer addition have been used to overcome the compatibility issues. To date, very limited work has been performed to study the mechanical and thermal properties of recycled PET reinforced with natural fibers. A through study of microscopic and macroscopic properties of PETr will ensure that the compound can be widely used in various outdoor applications, while it can be processed by a range of technological equipments, typically injection molding. Based on literature, several studies have been done on natural fiber/polymer composites but the effect of natural fiber additions to recycled PET received less attention. The present study is focused on the effect of date palm leaf fiber addition into PETr matrix, and the outcome will help to analyze the final composites and nanocomposites properties to be used and implemented as a replacement in applications where traditionally made of wood.

1. What are the benefits of using the surface treatment process and how can it enhance and change the properties of fibers and composites?
2. What improvements will be achieved by incorporating date palm leaf fibers (DPLF) into the PETr matrix in terms of mechanical and thermal properties?
3. Does the addition of Montmorillonite (MMT) helps enhancing the mechanical and thermal properties of the nanocomposites in order to be used in various outdoor applications where high stiffness is crucial?

1.3 Objectives of study

One of the most important aspects of composite developments is to achieve a good combination of mechanical properties and processability. As far as mechanical properties are concerned, the goal is to achieve a balance of stiffness, strength and toughness. The present work aims to develop plastic composites based on recycled polyethylene terephthalate (PETr) reinforced with date palm leaf fiber (DPLF). These composites have the potential to be used in indoor applications such as door frames,

trim and furniture as well as any other wood replacement materials. The main objectives can further be divided into:

- i. To determine the effect of chemical surface treatment on the thermal properties and chemical structure of the date palm leaf fiber.
- ii. To study the effect of date palm leaf fiber (DPLF) loading on mechanical and thermal properties as well as morphological (SEM) behavior of the recycled PET/DPLF composites.
- iii. To investigate the effect of montmorillonite (MMT) concentration on the mechanical and thermal properties of PETr/DPLF/MMT nanocomposites.

1.4 Scope of research

In order to achieve the goals of this research, the following procedures were carried out:

1. Sample preparation In this research project, sample preparation and blending was performed *via* melt extrusion. This includes:
 - i. Surface treating the date palm fiber.
 - ii. Blending of recycled PET with surface treated fibers by using a twin screw extruder in a single extrusion step.
 - iii. Blend fabrication into test specimens via injection molding according to ASTM test specimen.
2. Physical and mechanical analysis
 - i. Density measurement
 - ii. Tensile test
 - iii. Flexural test
 - iv. Izod impact test
3. Thermal properties analysis
 - i. Differential scanning calorimeter (DSC)
 - ii. Thermogravimetric analysis (TGA)
 - iii. Dynamic mechanical analysis (DMA)

4. Morphological study
 - i. Scanning electron microscopy (SEM)

REFERENCES

1. Corradini, E., Ito, E. N., Marconcini, J. M., Rios, C. T., Agnelli, J. A. M. and Mattoso, L. H. C. Interfacial behavior of composites of recycled poly(ethylene terephthalate) and sugarcane bagasse fiber. *Polymer Testing*, 2009. 28(2): 183–187.
2. Nikles, D. E. and Farahat, M. S. New Motivation for the Depolymerization Products Derived from Poly(Ethylene Terephthalate) (PET) Waste: a Review. *Macromolecular Materials and Engineering*, 2005. 290(1): 13–30.
3. Lepoittevin, B. and Roger, P. Poly (ethylene terephthalate). *Handbook of Engineering and Speciality Thermoplastics*, 2011: 97–126.
4. Spinace, M. and De Paoli, M. Characterization of poly (ethylene terephthalate) after multiple processing cycles. *Journal of Applied Polymer Science*, 2001. 80(1): 20–25.
5. Huang, J. J., Keskkula, H. and Paul, D. R. Rubber toughening of an amorphous polyamide by functionalized SEBS copolymers: morphology and Izod impact behavior. *Polymer*, 2004. 45(12): 4203–4215.
6. Oshinski, A., Keskkula, H. and Paul, D. Rubber toughening of polyamides with functionalized block copolymers: 2. Nylon-6, 6. *Polymer*, 1992. 33(2): 284–293.
7. Tjong, S. C. and Bao, S. P. Fracture toughness of high density polyethylene/SEBS-g-MA/montmorillonite nanocomposites. *Composites Science and Technology*, 2007. 67(2): 314–323.
8. Tjong, S. C., Bao, S. P. and Liang, G. D. Polypropylene/montmorillonite nanocomposites toughened with SEBS-g-MA: Structure-property relationship. *Journal of Polymer Science Part B: Polymer Physics*, 2005. 43(21): 3112–3126.
9. Chow, W. S. and Neoh, S. S. Mechanical, Morphological and Thermal Properties of Polycarbonate/SEBS-G-MA/Montmorillonite Nanocomposites. *Polymer-Plastics Technology and Engineering*, 2009. 49(1): 62–68.
10. Zhang, H., Guo, W., Yu, Y., Li, B. and Wu, C. Structure and properties

- of compatibilized recycled poly(ethylene terephthalate)/linear low density polyethylene blends. *European Polymer Journal*, 2007. 43(8): 3662–3670.
11. Yu, Z.-Z., Yang, M.-S., Dai, S.-C. and Mai, Y.-W. Toughening of recycled poly(ethylene terephthalate) with a maleic anhydride grafted SEBS triblock copolymer. *Journal of Applied Polymer Science*, 2004. 93(3): 1462–1472.
 12. Zhang, Y., Zhang, H., Ni, L., Zhou, Q., Guo, W. and Wu, C. Crystallization and Mechanical Properties of Recycled Poly (ethylene terephthalate) Toughened by Styrene–Ethylene/Butylenes–Styrene Elastomer. *Journal of Polymers and the Environment*, 2010. 18(4): 647–653.
 13. Kaddami, H., Dufresne, A., Khelifi, B., Bendahou, A., Taourirte, M., Raihane, M., Issartel, N., Sautereau, H., Gérard, J.-F. and Sami, N. Short palm tree fibers – Thermoset matrices composites. *Composites Part A: Applied Science and Manufacturing*, 2006. 37(9): 1413–1422.
 14. Oksman, K. Mechanical properties of natural fibre mat reinforced thermoplastic. *Applied Composite Materials*, 2000. 7(5): 403–414.
 15. Mwaikambo, L. Y. and Ansell, M. P. Chemical modification of hemp, sisal, jute, and kapok fibers by alkalization. *Journal of Applied Polymer Science*, 2002. 84(12): 2222–2234.
 16. Najafi, S. K., Hamidinia, E. and Tajvidi, M. Mechanical properties of composites from sawdust and recycled plastics. *Journal of Applied Polymer Science*, 2006. 100(5): 3641–3645.
 17. Tajvidi, M., Falk, R. H. and Hermanson, J. C. Effect of natural fibers on thermal and mechanical properties of natural fiber polypropylene composites studied by dynamic mechanical analysis. *Journal of Applied Polymer Science*, 2006. 101(6): 4341–4349.
 18. Herrera-Franco, P. J. and Valadez-Gonzalez, A. A study of the mechanical properties of short natural-fiber reinforced composites. *Composites Part B: Engineering*, 2005. 36(8): 597–608.
 19. Al-Kaabi, K., Al-Khanbashi, A. and Hammami, A. Date palm fibers as polymeric matrix reinforcement: DPF/polyester composite properties. *Polymer Composites*, 2005. 26(5): 604–613.
 20. Pavithran, C., Mukherjee, P. S., Brahmakumar, M. and Damodaran, A. D. Impact properties of natural fibre composites. *Journal of Materials Science Letters*, 1987. 6(8): 882–884.
 21. Luo, S. and Netravali, A. Mechanical and thermal properties of

- environmentfriendly “green” composites made from pineapple leaf fibers and poly (hydroxybutyratecovalerate) resin. *Polymer Composites*, 1999. 20(3): 367–378.
22. Soleimani, M., Tabil, L., Panigrahi, S. and Opoku, A. The Effect of Fiber Pretreatment and Compatibilizer on Mechanical and Physical Properties of Flax Fiber-Polypropylene Composites. *Journal of Polymers and the Environment*, 2008. 16(1): 74–82.
 23. Al-Khanbashi, A., Al-Kaabi, K. and Hammami, A. Date palm fibers as polymeric matrix reinforcement: Fiber characterization. *Polymer Composites*, 2005. 26(4): 486–497.
 24. Alawar, A., Hamed, A. and Al-Kaabi, K. Characterization of treated date palm tree fiber as composite reinforcement. *Composites Part B: Engineering*, 2009. 40(7): 601–606.
 25. Chilton, T., Burnley, S. and Nesaratnam, S. A life cycle assessment of the closed-loop recycling and thermal recovery of post-consumer PET. *Resources, Conservation & Recycling*, 2010. 54(12): 1241–1249.
 26. Donnini, M. Recyclability of PET from virgin resin. *Materials Research*, 1999.
 27. Baliga, S. and Wong, W. Depolymerization of poly (ethylene terephthalate) recycled from postconsumer softdrink bottles. *Journal of Polymer Science Part A: Polymer Chemistry*, 2002. 27(6): 2071–2082.
 28. Chen, C. Study of glycolysis of poly (ethylene terephthalate) recycled from postconsumer softdrink bottles. III. Further investigation. *Journal of Applied Polymer Science*, 2003. 87(12): 2004–2010.
 29. Chen, C., Chen, C., Lo, Y., Mao, C. and Liao, W. Studies of glycolysis of poly (ethylene terephthalate) recycled from postconsumer softdrink bottles. I. Influences of glycolysis conditions. *Journal of Applied Polymer Science*, 2001. 80(7): 943–948.
 30. Chen, C., Chen, C., Lo, Y., Mao, C. and Liao, W. Studies of glycolysis of poly (ethylene terephthalate) recycled from postconsumer softdrink bottles. II. Factorial experimental design. *Journal of Applied Polymer Science*, 2001. 80(7): 956–962.
 31. Torres, N., Robin, J. and Boutevin, B. Study of thermal and mechanical properties of virgin and recycled poly (ethylene terephthalate) before and after injection molding. *European Polymer Journal*, 2000. 36(10): 2075–2080.

32. Wilfong, D. L., Hiltner, A. and Baer, E. Toughening of polyester resins through blending with polyolefins. *Journal of Materials Science*, 1986. 21(6): 2014–2026.
33. Sammon, C., Yarwood, J. and Everall, N. An FT-IR study of the effect of hydrolytic degradation on the structure of thin PET films. *Polymer Degradation and Stability*, 2000. 67(1): 149–158.
34. Golike, R. C. and Lasoski, S. W. Kinetics of hydrolysis of polyethylene terephthalate films. *Journal of Physical Chemistry*, 1960. 64(7): 895–898.
35. Sinha Ray, S. and Bousmina, M. Effect of organic modification on the compatibilization efficiency of clay in an immiscible polymer blend. *Macromolecular rapid communications*, 2005. 26(20): 1639–1646.
36. Ou, C. F., Ho, M. and Lin, J. The nucleating effect of montmorillonite on crystallization of PET/montmorillonite nanocomposite. *Journal of Polymer Research*, 2003. 10(2): 127–132.
37. Wan, T., Chen, L., Chua, Y. C. and Lu, X. Crystalline morphology and isothermal crystallization kinetics of poly(ethylene terephthalate)/clay nanocomposites. *Journal of Applied Polymer Science*, 2004. 94(4): 1381–1388.
38. Sánchez Solís, A., Romero-Ibarra, I., Estrada, M. R., Calderas, F. and Manero, O. Mechanical and rheological studies on polyethylene terephthalate-montmorillonite nanocomposites. *Polymer Engineering and Science*, 2004. 44(6): 1094–1102.
39. Wang, Y., Gao, J., Ma, Y. and Agarwal, U. Study on mechanical properties, thermal stability and crystallization behavior of PET/MMT nanocomposites. *Composites Part B: Engineering*, 2006. 37(6): 399–407.
40. Bizarria, M., Giraldi, A., de Carvalho, C., Velasco, J., d'Ávila, M. and Mei, L. Morphology and thermomechanical properties of recycled PET–organoclay nanocomposites. *Journal of Applied Polymer Science*, 2007. 104(3): 1839–1844.
41. Giraldi, A. L. F. d. M., Bizarria, M. T. M., Silva, A. A., Velasco, J., d'Ávila, M. A. and Mei, L. Effects of extrusion conditions on the properties of recycled poly(ethylene terephthalate)/nanoclay nanocomposites prepared by a twin-screw extruder. *Journal of Applied Polymer Science*, 2008. 108(4): 2252–2259.
42. Davis, C. H., Mathias, L. J., Gilman, J. W., Schiraldi, D. A., Shields, J. R., Trulove, P., Sutto, T. E. and DeLong, H. C. Effects of melt-processing

- conditions on the quality of poly(ethylene terephthalate) montmorillonite clay nanocomposites. *Journal of Polymer Science Part B: Polymer Physics*, 2002. 40(23): 2661–2666.
43. Ke, Y., Long, C. and Qi, Z. Crystallization, properties, and crystal and nanoscale morphology of PET–clay nanocomposites. *Journal of Applied Polymer Science*, 1999. 71(7): 1139–1146.
 44. Ke, Y.-C., Yang, Z.-B. and Zhu, C.-F. Investigation of properties, nanostructure, and distribution in controlled polyester polymerization with layered silicate. *Journal of Applied Polymer Science*, 2002. 85(13): 2677–2691.
 45. Eichhorn, S., Dufresne, A., Aranguren, M., Marcovich, N., Capadona, J., Rowan, S., Weder, C., Thielemans, W., Roman, M. and Renneckar, S. Review: current international research into cellulose nanofibres and nanocomposites. *Journal of Materials Science*, 2010. 45(1): 1–33.
 46. Mohantya, A. K., Misra, M. and Hinrichsen, G. Biofibres, biodegradable polymers and biocomposites. 2011: 1–24.
 47. Taj, S., Munawar, M. and Khan, S. Natural fiber-reinforced polymer composites. *PROCEEDINGS-PAKISTAN ACADEMY OF SCIENCES*, 2007. 44(2): 129.
 48. Kalia, S., Kaith, B. S. and Kaur, I. Pretreatments of natural fibers and their application as reinforcing material in polymer composites-A review. *Polymer Engineering and Science*, 2009. 49(7): 1253–1272.
 49. Oksman, K., Mathew, A., Långström, R., Nyström, B. and Joseph, K. The influence of fibre microstructure on fibre breakage and mechanical properties of natural fibre reinforced polypropylene. *Composites Science and Technology*, 2009. 69(11-12): 1847–1853.
 50. Monteiro, S. N., Terrones, L. A. H. and D’Almeida, J. R. M. Mechanical performance of coir fiber/polyester composites. *Polymer Testing*, 2008. 27(5): 591–595.
 51. Bendahou, A., Kaddami, H., Sautereau, H., Raihane, M., Erchiqui, F. and Dufresne, A. Short Palm Tree Fibers Polyolefin Composites: Effect of Filler Content and Coupling Agent on Physical Properties. *Macromolecular Materials and Engineering*, 2008. 293(2): 140–148.
 52. John, M. J. and Anandjiwala, R. D. Recent developments in chemical modification and characterization of natural fiber-reinforced composites. *Polymer Composites*, 2008. 29(2): 187–207.

53. Wambua, P., Ivens, J. and Verpoest, I. Natural fibres: can they replace glass in fibre reinforced plastics? *Composites Science and Technology*, 2003. 63(9): 1259–1264.
54. Hill, C. and Abdul Khalil, H. Effect of fiber treatments on mechanical properties of coir or oil palm fiber reinforced polyester composites. *Journal of Applied Polymer Science*, 2000. 78(9): 1685–1697.
55. Nair, K., Diwan, S. and Thomas, S. Tensile properties of short sisal fiber reinforced polystyrene composites. *Journal of Applied Polymer Science*, 1996. 60(9): 1483–1497.
56. Keener, T. J., Stuart, R. K. and Brown, T. K. Maleated coupling agents for natural fibre composites. *Composites Part A: Applied Science and Manufacturing*, 2004. 35(3): 357–362.
57. Hristov, V., Krumova, M., Vasileva, S. and Michler, G. Modified polypropylene wood flour composites. II. Fracture, deformation, and mechanical properties. *Journal of Applied Polymer Science*, 2004. 92(2): 1286–1292.
58. Guo, C. G. and Wang, Q. W. Compatibilizing Effect of Maleic Anhydride Grafted Styrene-Ethylene-Butylene-Styrene (MAH-g-SEBS) on the Polypropylene and Wood Fiber Composites. *Journal of Reinforced Plastics and Composites*, 2007. 26(17): 1743–1752.
59. Sreekala, M., Kumaran, M., Joseph, S., Jacob, M. and Thomas, S. Oil palm fibre reinforced phenol formaldehyde composites: Influence of fibre surface modifications on the mechanical performance. *Applied Composite Materials*, 2000. 7: 295–329.
60. Valadez-Gonzalez, A., Cervantes-Uc, J., Olayo, R. and Herrera-Franco, P. Chemical modification of henequen fibers with an organosilane coupling agent. *Composites Part B: Engineering*, 1999. 30(3): 321–331.
61. Saheb, D. and Jog, J. Natural fiber polymer composites: a review. *Advances in Polymer Technology*, 1999. 18(4): 351–363.
62. Sanadi, A. R., Prasad, S. V. and Rohatgi, P. K. Sunhemp fibre-reinforced polyester. *Journal of Materials Science*, 1986. 21(12): 4299–4304.
63. Son, J., Yang, H. and Kim, H. Physico-mechanical properties of paper sludge-thermoplastic polymer composites. *Journal of Thermoplastic Composite Materials*, 2004. 17(6): 509–522.
64. Cao, Y., Shibata, S. and Fukumoto, I. Mechanical properties of biodegradable

- composites reinforced with bagasse fibre before and after alkali treatments. *Composites Part A: Applied Science and Manufacturing*, 2006. 37(3): 423–429.
65. Huda, M. S., Drzal, L. T., Misra, M., Mohanty, A. K., Williams, K. and Mielewski, D. F. A Study on Biocomposites from Recycled Newspaper Fiber and Poly(lactic acid). *Industrial & Engineering Chemistry Research*, 2012. 44(15): 5593–5601.
 66. Wielage, B., Lampke, T., Marx, G., Nestler, K. and Starke, D. Thermogravimetric and differential scanning calorimetric analysis of natural fibres and polypropylene. *Thermochimica Acta*, 1999. 337(1): 169–177.
 67. Balakrishnan, H., Hassan, A. and Wahit, M. U. Mechanical, thermal, and morphological properties of polylactic acid/linear low density polyethylene blends. *Journal of Elastomers and Plastics*, 2010. 42(3): 223–239.
 68. Liu, Z., Chen, K. and Yan, D. Nanocomposites of poly(trimethylene terephthalate) with various organoclays: morphology, mechanical and thermal properties. *Polymer Testing*. 23(3): 323–331.
 69. Boon, J., Challa, G. and Van Krevelen, D. Crystallization kinetics of isotactic polystyrene. I. Spherulitic growth rate. *Journal of Polymer Science Part A2: Polymer Physics*, 1968. 6(10): 1791–1801.
 70. Balakrishnan, H., Hassan, A. and Isitman, N. On the use of magnesium hydroxide towards halogen-free flame-retarded polyamide-6/polypropylene blends. *Polymer Degradation and ...*, 2012.
 71. Feijoo, J., Cabedo, L., Gimenez, E., Lagaron, J. and Saura, J. Development of amorphous PLA-montmorillonite nanocomposites. *Journal of Materials Science*, 2005. 40(7): 1785–1788.
 72. Pluta, M. Melt compounding of polylactide/organoclay: Structure and properties of nanocomposites. *Journal of Polymer Science Part B: Polymer Physics*, 2006. 44(23): 3392–3405.
 73. Sinha Ray, S. and Okamoto, M. Polymer/layered silicate nanocomposites: a review from preparation to processing. *Progress in Polymer Science*, 2003. 28(11): 1539–1641.
 74. Wu, T.-M. and Wu, C.-Y. Biodegradable poly(lactic acid)/chitosan-modified montmorillonite nanocomposites: Preparation and characterization. *Polymer Degradation and Stability*, 2006. 91(9): 2198–2204.
 75. English, A. Macromolecular dynamics in solid poly (ethylene terephthalate):

- proton and carbon-13 solid-state NMR. *Macromolecules*, 1984. 17(10): 2182–2192.
76. Maxwell, A., Monnerie, L. and Ward, I. Secondary relaxation processes in polyethylene terephthalate–additive blends: 2. Dynamic mechanical and dielectric investigations. *Polymer*, 1998. 39(26): 6851–6859.
77. Mackintosh, A. and Liggat, J. Dynamic mechanical analysis of poly (trimethylene terephthalate)—A comparison with poly (ethylene terephthalate) and poly (ethylene naphthalate). *Journal of Applied Polymer Science*, 2004. 92(5): 2791–2796.
78. Thompson, A. B. and Woods, D. W. The transitions of polyethylene terephthalate. *Transactions of the Faraday Society*, 1956. 52: 1383.